AFI SAPO-5 Al(49),P(35),Si(16)

Contributed by David Young

Verified by R. Borade and S. Schunk

Type Material $[Al_{11.8}P_{9.4}Si_{3.8}O_{48}] : rR : wH_2O$

Method D. Young, M. F. Davis [1] [a]

Batch Composition 0.15 cHA : 0.25 $[(CH_3)_2CHO]_3Al$: 0.25 H_3PO_4 : 13 H2O 0.5 SiO_2

Source Materials

deionized water
aluminum isopropoxide (Aldrich, 98+%)
phosphoric acid (Aldrich A.C.S. grade, 85% H_3PO_4)
cyclohexylamine (cHA)(Aldrich, 99+%)
silica sol (Dupont Ludox AS-40, 40% SiO_2)

Batch Preparation (for 1 g product) [b]

(1) [4 g water + 1.44 g aluminum isopropoxide]; make slurry
(2) [1.2 g water + 0.81 g phosphoric acid]; mix
(3) [(1) + (2)]; add diluted phosphoric acid dropwise to the aluminum isopropoxide slurry. Stir and age the gel for one hour to ensure homogeneity
(4) [(3) + 0.42 g cHA]; add cHA dropwise. A viscous gel results. Stir and age for 90 minutes
(5) [(4) + 2.13 g silica sol]; check to see that gel (4) is homogeneous, then add silica sol and stir for 10 minutes [c]

Crystallization

Vessel: 15 mL Teflon-lined autoclave [d]
Temperature: 200˚C [e]
Time: 3 hours [f]
Agitation: none

Product Recovery

(1) Remove reactor from the oven and quench cool
(2) Transfer product from the liner to a beaker with a wash bottle [g]
(3) Slurry with 50 mL deionized water. Allow the SAPO-5 crystallites to settle and decant off the suspended impurities. Repeat twice

(4) Filter off product on a Buechner funnel. Wash copiously with water. Air dry
(5) Yield: 1 g (Approximately 85% with respect to phosphoric acid)

Product Characterization

XRD: high purity SAPO-5 free from amorphous or crystalline impurities (SAPO-44) [d]
Elemental Analysis: $Al_{0.49}P_{0.35}Si_{0.16}O2$ [h]

Crystal size and habit: spherical or hexagonal aggregates, average size of 20 μm

References

[1] D. Young, M. E. Davis, Zeolites 11 (1991) 277
[2] S. T. Wilson, B. M. Lok, E. M. Flanigen, US Patent 4 310 440 (1982)
[3] J. A. Martens, C. Janssens, P. J. Grobet, H. K. Beyer, P. A. Jacobs, Stud. Surf. Sci. Catal. 49A (1989) 215

Notes

a. Developed from S. T. Wilson, et al., [2] and from J. A. Martens, et al., [3]
b. When handling small quantities of polar liquids, the use of glassware which has been pretreated with dichiorodimethylsilane is recommended. This ensures a clean transfer of reagents.
c. Failure to mix the reagents in this order will result in different products, i.e., the silicoaluminophosphate analogue of quartz/berlinite.
d. Due to the rapid crystallization of the SAPO-5 product and its propensity to transform to SAPO-44 upon synthesis over-run, small reactors with a narrow aspect ratio are recommended. This synthesis is recommended for a 15 mL capacity reactor. It can be scaled up, but the gel should be split between small reactors. Even with 45 mL reactors, impure product will result.
e. Place autoclave on a rack in a forced convection oven at 200˚C.
f. A four-hour synthesis time results in SAPO-44 impurities: a two-hour reaction yields an amorphous gel. Deliberate over-run of one week will yield an excellent SAPO-44.
g. The pH of the synthesis mother liquor will peak at close to 10, which coincides with the crystallization of the SAPO-5.
h. This indicates that the main mode of T-atom substitution is silicon for phosphorous. However, surface analysis reveals significant silicon enrichment with strong evidence for silica islanding by Si-O-Si substitution for Al-O-P. ^{29}Si NMR: P-substitution peak at -90 ppm and Si-O-Si peak at -110 ppm.